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Low Temperature Thermal Reduction of Graphene Oxide

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Graphene oxide (GO), an oxidized form of graphene, has attracted considerable interests as it can be easily synthesized, processed, and thermally reduced to graphene-like sheet [1]. However, through the existing removal methods, all of the reduced GO (rGO) products still contain residual oxygens and other structural defect [2,3]. This problem must be solved if we want to be able to design and tailor the properties of reduced GO. To do so, we should have better understanding of two topics: (1) what are the reduction mechanisms; (2) how to control the oxidation degree. Thus, we can not only obtain graphene-like materials with controllable functionalities in an economically effective manner, but also fulfil different specific application requirements. Choosing proper fabricating method and reducing process are two key factors for future industrial production. Hummers' method as an efficient synthetic protocol for GO is mostly widely used. Since the chemical structure and the kinetics of functional groups are still unclear, it is important to investigate the formation process of the main product and by-products obtained by Hummers' method, and to reveal the mechanism of reduction for large-scale production.

In this study, we focus on studying the decomposition mechanism of GO at relatively low temperatures (from 120 to 200 °C) (close to industrial production temperature) for different annealing duration (0.5, 1, 3, 7 and 24 hours). We find that the reduction process of multilayer GO can be divided into three steps from the DSC-TGA results (Fig.1a). Step 3 (temperature range: 210-300 °C), is an endothermic reaction, which relates to the decomposition of the carbon plane and the derivatives of organics, as well as SO₂, instead of water, CO₂ or CO (Fig 1b). Based on these findings, kinetic and elemental measurements were carried out by using XPS and FTIR-GC-MS in order to understand the kinetics and chemical mechanisms of low temperature GO reduction.

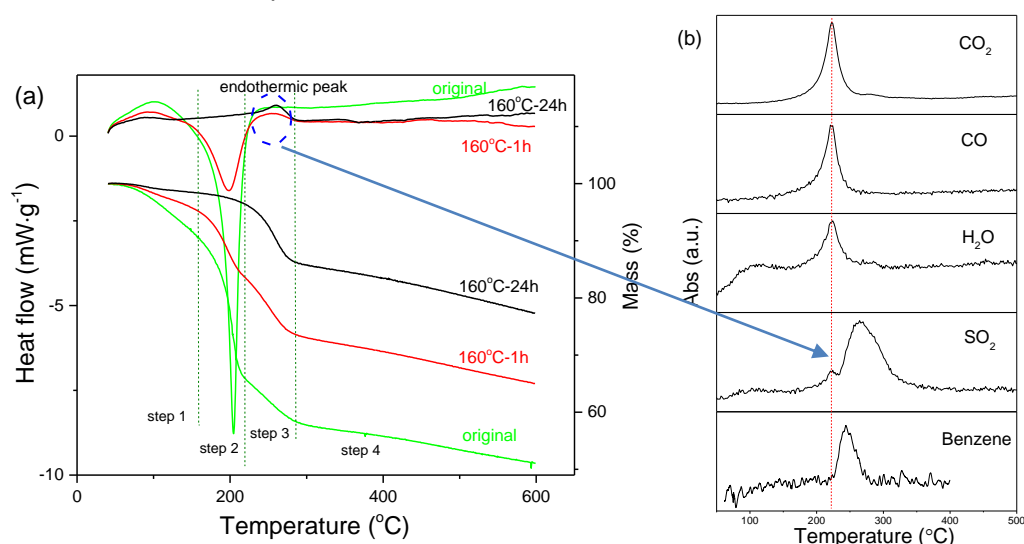


Fig. 1. (a) DSC-TGA data for both original and annealed GO samples: (160°C-1h and 160°C-24h); (b) FTIR-GC-MS data for the by-products of the reduction process.

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